Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

N,*N*:*N*′,*N*′-Bis(2,2′-dihydroxybiphenyl-3,3′-dimethylidene)benzene-1,2-diamine dimethyl sulfoxide tetrasolvate

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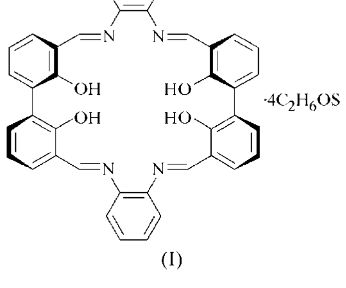
Received 15 February 2005
Accepted 6 April 2005
Online 20 May 2005

The title compound, 8,15,28,35-tetraazaheptacyclo[35.3.1.1^{2,6}.1^{17,21}.1^{22,26}.0^{9,14}.0^{29,34}]tetraconta-1(41),2,4,6(42),7,9,11,13,15,17,19,21(43),22,24,26(44),27,29,31,33,35,37,39-docosaene-41,42,43,44-tetrol dimethyl sulfoxide tetrasolvate, $C_{40}H_{28}N_4O_4 \cdot 4C_2H_6OS$, adopts a chair-shaped C_{2h} symmetric conformation with crystallographically imposed inversion symmetry. Four intramolecular hydrogen bonds are observed between phenol O and imine N atoms.

Comment

Our ongoing studies of the coordination chemistry of macrocyclic ligands (Kühn *et al.*, 2001) and metal–functional group co-operation (Kövari & Krämer, 1996; Plitt *et al.*, 2004) require the synthesis and modelling of known and novel cyclic compounds. The Schiff base macrocycle in the title compound, (I), attracts attention since it might be considered as a dimeric C—C-coupled form of the well described ligand *N*,*N*′-bis(salicylidene)benzene-1,2-diamine (salphen). Biphenyl-derived Schiff base macrocycle (I) is analogous to the binaphthyl-derived compounds prepared by Brunner & Schiesling (1994) and Zhang *et al.* (2001). However, Zhang and co-workers describe the formation of a polymer when condensing the biphenyl-derived dicarbaldehyde with benzene-1,2-diamine in ethanol. We report here the first synthesis of (I), which can be obtained in good yield when acetonitrile is used as solvent. To the best of our knowledge, no structural data have previously been published either for (I) or for its binaphthyl derivative and analogues containing different 1,2-diamine moieties.



(I)

The molecular structure of (I) is shown in Fig. 1. The compound crystallizes from dimethyl sulfoxide (DMSO) solution. X-ray diffraction analysis reveals one molecule of (I) and four molecules of DMSO per unit cell. In the crystal structure, the molecule shows a chair-shaped C_{2h} symmetric conformation with crystallographically imposed inversion symmetry (symmetry code: $2 - x, 1 - y, 2 - z$). Selected bond distances and angles (Table 1) are typical and do not differ from those observed for salphen (Bresciani Pahor *et al.*, 1976).

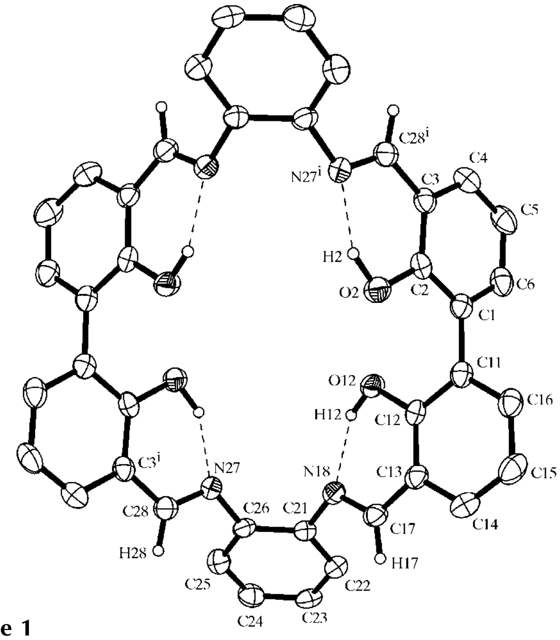


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary size. DMSO molecules and aromatic H atoms have been omitted for clarity. Atoms without labels or marked with a superscript ‘i’ are at the symmetry position ($2 - x, 1 - y, 2 - z$).

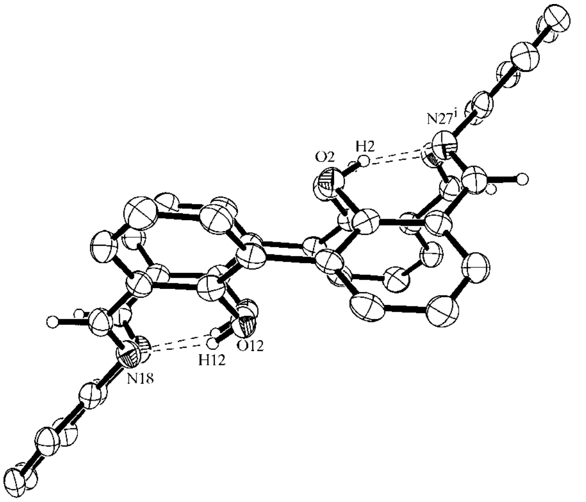


Figure 2
An alternative view of (I), illustrating the chair conformation.

DOI: 10.1107/S010827010501022X

For the phenol rings, the attached O (O2 and O12) and C atoms (C17 and C28^{i}), and for the phenylenediamine rings, the attached N atoms (N18 and N27) deviate from the ring planes by only 0.04, 0.14 and 0.07 Å (mean values), respectively. The imine unit (C—C═N) and the phenol rings are almost coplanar, with interplanar angles of 7.0 (4) and 7.4 (4)°, respectively, whereas on the other side, the imine unit (C—N═C) and the phenylenediamine rings deviate from coplanarity by 47.6 (4) and 54.8 (4)°, respectively. As expected, the biphenylene unit is not planar. The two aromatic rings are twisted relative to each other by an angle of 76.8 (4)° (Fig. 2).

Schiff base macrocycle (I) contains four intramolecular O—H···N hydrogen bonds between the phenol O atoms and the imine N atoms (Table 2). No notable intermolecular interactions between the molecules of (I) or interactions involving the solvent molecules were found.

Experimental

Schiff base macrocycle (I) was prepared using a procedure similar to that described for the analogous binaphthyl derivative (Brunner & Schiesling, 1994; Zhang *et al.*, 2001). 2,2′-Dihydroxybiphenyl-3,3′-dicarbaldehyde (484 mg, 2 mmol) (Yardley & Fletcher, 1976; Zhang *et al.*, 2001) and benzene-1,2-diamine (216 mg, 2 mmol) were dissolved in dry acetonitrile (20 ml) and refluxed overnight. The precipitate which formed was filtered off, washed with acetonitrile (3 × 1 ml) and dried *in vacuo*, yielding a pale-yellow solid (70% yield). ^{1}H NMR (200 MHz, DMSO-d_6): δ 7.56–7.59 (*m*, 6H, H$_{ar}$), 8.26 (*s*, 3H, CHN), 14.47 (*s*, 3H, OH); IR (KBr, ν, cm^{-1}): 1617 (*s*), 1582 (*m*), 1437 (*m*), 1212 (*m*), 826 (*m*), 779 (*m*), 747 (*s*). HRMS (ESI+): *m*/*z*, calculated for $C_{40}H_{29}N_4O_4$ ($[M+H]^+$): 629.2189; found: 629.2188. Elemental analysis (%) calculated for $C_{40}H_{28}N_4O_4\cdot 2CH_3CN$: C 74.35, H 4.82, N 11.82; found: C 74.29, H 4.78, N 11.89. Single crystals were obtained by slow cooling of a saturated solution of (I) in DMSO.

Crystal data

$C_{40}H_{28}N_4O_4\cdot 4C_2H_6OS$
$M_r = 941.18$
Triclinic, $P\overline{1}$
$a = 10.4876$ (6) Å
$b = 11.2572$ (7) Å
$c = 12.4998$ (8) Å
$\alpha = 99.603$ (1)°
$\beta = 110.874$ (2)°
$\gamma = 112.700$ (2)°
$V = 1190.15$ (13) Å^3
$Z = 1$
$D_x = 1.313$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1790 reflections
$\theta = 1.9$–27.5°
$\mu = 0.26$ mm^{-1}
$T = 200$ (2) K
Polyhedron, yellow
0.12 × 0.10 × 0.08 mm

Table 1
Selected geometric parameters (Å, °).

C2—O2	1.362 (3)	C26—N27	1.422 (3)
C2—C3	1.412 (4)	N27—C28	1.286 (3)
C3—C28^{i}	1.458 (4)		
O2—C2—C3	120.6 (3)	C28—N27—C26	118.4 (2)
C2—C3—C28^{i}	121.5 (3)	N27—C28—C3^{i}	122.9 (3)
C21—C26—N27	118.7 (2)		
O2—C2—C3—C28^{i}	3.8 (4)	C25—C26—N27—C28	55.7 (4)
C2—C1—C11—C12	78.5 (3)	C21—C26—N27—C28	−126.5 (3)
O12—C12—C13—C17	6.2 (4)	C26—N27—C28—C3^{i}	−175.5 (2)
C12—C13—C17—N18	−6.5 (4)	C2—C3—C28^{i}—N27^{i}	−6.8 (4)

Symmetry code: (i) $2 - x, 1 - y, 2 - z$.

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.886$, $T_{max} = 0.980$
10 638 measured reflections
4343 independent reflections
2489 reflections with $I > 2\sigma(I)$
$R_{int} = 0.064$
$\theta_{max} = 25.3°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.112$
$S = 0.99$
4343 reflections
315 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.0226P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N27^{i}	0.96 (3)	1.75 (4)	2.626 (3)	150 (2)
O12—H12···N18	0.90 (4)	1.80 (4)	2.603 (3)	147 (2)

Symmetry code: (i) $2 - x, 1 - y, 2 - z$.

Hydroxyl H atoms were refined isotropically (H1 and H12 on O2 and O12). All other H atoms were included in the structural model at geometrically calculated positions, with C—H distances in the range 0.95–0.98 Å, and refined riding on their parent C atom, with $U_{iso}(H) = 1.2$ or $1.5U_{eq}(C)$. Two DMSO solvent molecules were found in the asymmetric unit. One was exactly located, while the other was found in two alternative orientations (93:7 occupancy). Both instances of this disordered DMSO molecule, as well as the proper DMSO molecule, were constrained to have similar chemically equivalent distances and angles.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1234). Services for accessing these data are described at the back of the journal.

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